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Studies on barium ion bound guar sample: Preparation and characterization

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Abstract
Guargum is a water soluble natural polymer and is used in industries like paper industry. When guargum is grafted with polyacrylamide side chains, its biodegradation resistance increases. Guargum – graft – acrylamide binds Y^{3+}, Ba^{2+} and Cu^{2+} by – COOH groups of side chains at higher pH. Study of binding Y^{3+} and Cu^{2+} by guargum itself has been reported earlier. In this work, study of binding Ba^{2+} by guar sample has been done by SEM, EDS and IR spectra. These techniques support association of polymeric chains of guar sample by Ba^{2+} ions at high pH and also binding of Ba^{2+} ions by guar sample by the use of some – C-OH groups of guar sample.

Keywords: Guargum, barium ion, scanning electron microscope, electron diffraction, ion binding.

1. Introduction
Recently because of economic reason, petroleum based polymers may not be very much suitable for industrial applications. Guargum is a water soluble natural polymer and has good industrial use [1]. Its structure is relatively known (fig.1). To improve biodegradation resistance of guargum, polyacrylamide side chains can be grafted on C – 2 or C – 3 position of guargum to get guargum – graft – acrylamide (G – g – Am) [2]. It has been found that G – g – Am is an efficient flocculent for metallic ions [2]. So this graft copolymer may be used for preparation of $Y_1\ Ba_2\ Cu_3\ O_{7-x}$ ceramic oxide superconductor by polymeric precursor technique [3]. Initial step of it is to study the ion binding behavior of G – g – Am. It has been found that aqueous solution of G – g – Am binds Y^{3+}, Ba^{2+} and Cu^{2+} in different pH range and - COO^- groups from side chains of G – g Am at higher pH are utilized for binding Y^{3+} or Ba^{2+} or Cu^{2+} and has been reported [4]. From transmission electron diffraction (TED) study it was possible to understand that G – g – Am contains structural regularity but this structural regularity is absent in TED patterns for Y^{3+} ion bound G – g – Am (G – g – Am – Y^{3+}), Ba^{2+} ion bound G – g – Am (G – g – Am – Ba^{2+}) and Cu^{2+} ion bound G – g – Am (G – g – Am – Cu^{2+}) [4]. Structural regularity in G – g – Am may arise from polar – polar interaction between –OH groups in guargum backbones of G – g – Am chains. Any breakage in this interaction may be because of involvement of - OH groups of backbone of G – g – Am in binding polyvalent metal ions. This motivated research on binding of Y^{3+}, Ba^{2+} and Cu^{2+} by aqueous solution of guargum at higher pH. It has been already reported that aqueous solution of guargum when is treated with Y^{3+} or Cu^{2+} and pH is raised, a distinct mass separates and the resultant mass has been studied [5]. In this present investigation, whether aqueous solution of industrial guar sample is binding Ba^{2+} or not, has been studied. It has been found that aqueous solution of guar sample when is treated with Ba^{2+} and pH is raised, a white mass floats upon standing which is used for detailed study by SEM, EDS and IR techniques.

2. Materials and Methods
2.1. Guar sample
Guar sample used in this work is obtained from the Paper Industry: ITC Limited, Paper Boards and Specialty Papers Division, Unit: Tribeni, Hooghly, West Bengal, India. It was supplied from Sumita Hydro Colloid Limited, Jodhpur, India.

2.2. Ba $(NO_3)_2$ solution
Ba $(NO_3)_2$ solution has been prepared by dissolving solid barium nitrate in distilled water to get approximately 0.1 (M) solution (saturated solution).

2.3 Guar – Ba^{2+} sample

To prepare barium ion bound guar (Guar – Ba^{2+} sample), 100 ml approximately 1wt% guar sample is mixed with 10ml $Ba(NO_3)_2$ solution. 20 ml 60% NaOH solution is added. Mixture becomes light yellow. Mixture is allowed to stand overnight. A white portion floats over light yellow mixture. 30ml methanol is added and allowed to stand for 10mins. A Comparatively tight white mass separates which is taken out by glass rod and kept in another beaker containing 10ml methyl alcohol. After some time, methanol is removed by decantation and white tight mass is washed with 5ml methanol for three times in the same way. Then it is washed with 15 ml distilled water for five times. Then it is kept in the oven for drying for IR spectral study and electron diffraction study.

2.4. Barium hydroxide

10 ml barium nitrate solution is mixed with 10 ml 60% NaOH. Mixture is shaken and filtered (by a filter paper) to collect Ba $(OH)_2$ precipitate. This precipitate is dried in an oven for IR spectral study.

2.5. SEM

Scanning Electron Microscopic (SEM) study has been carried out using guar sample and guar – Ba^{2+} sample after gold coating. Gold coating has been done by SPUTTERING technique. Instrument used for SEM study is of JEOL company of Japan and has model number JSM – 5800 (SCANNING MICROSCOPE).

2.6. EDS

Electron Diffraction Study (EDS) has been carried out using INCA Software. Name of the company is OXFORD INSTRUMENTS with model number LINK ISI 300.

2.7 IR

FTIR instrument has been used for guar sample, guar – Ba^{2+} sample and barium hydroxide. Model number of the instrument is NICOLET 6700. KBr pellet technique has been adopted for IR spectral study. Spectral range covered was 4000 cm^{-1} to 400 cm^{-1}.

3. Results and Discussion

3.1 SEM

SEM images for guar sample and guar – Ba^{2+} sample are shown in fig. 2 and fig. 3 respectively. SEM image for guar sample shows discrete particles but SEM image for guar – Ba^{2+} sample shows much more continuous mass. This is indicating association of polymeric chains by Ba^{2+} ions in guar –Ba^{2+} sample.

3.2 EDS

Bulk EDS for guar sample (Table -1) (fig.4) is showing presence of C,O and trace amount of Ca (hydrogen can not be detected by EDS). Bulk EDS study for guar – Ba^{2+} sample (Table -2) (fig.5) shows presence of approximately 30% barium which is a strong evidence of binding Ba^{2+} ions by guar sample to get guar – Ba^{2+} sample.

Table 1: Bulk EDS study for guar sample

Element	Weight%	Atomic%
C	42.81	50.80
O	53.93	48.04
Ca	3.26	1.16
Total	100.00	

Table 2: Bulk EDS study for guar – Ba^{2+} sample

Element	Weight %	Atomic%
C	28.93	48.95
O	33.99	43.17
Ca	6.66	3.38
Ba	30.42	4.50
Total	100.00	

3.3 IR

IR spectra for guar sample, guar – Ba $^{2+}$ sample and $Ba(OH)_2$ are shown in figs.6-8 respectively. Peak position at 3370.87 cm^{-1} in the IR spectra for guar sample can be attributed to –OH stretching vibration of –C-O-H (hydrogen bonded). This peak has been shifted to 3424.49 cm^{-1} in the IR spectra for guar – Ba^{2+} sample. This is probably because of breaking of some hydrogen bonds in guar sample due to involvement of some –C-OH of guar sample in bindings Ba^{2+} ions to get guar –Ba^{2+} sample.

Strong peak at 856.36 cm^{-1} in the IR spectra for $Ba(OH)_2$ can be attributed to M-O-H bending vibration. Most interesting point to note in this study is the absence of this peak in the IR spectra for guar sample as well as in the IR spectra for guar-Ba^{2+} sample. This is the strongest point of this present investigation.

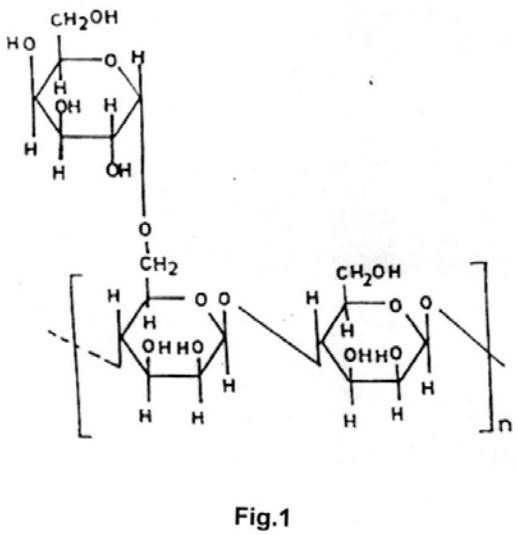

Fig.1

Fig 1: Structure of guargam.

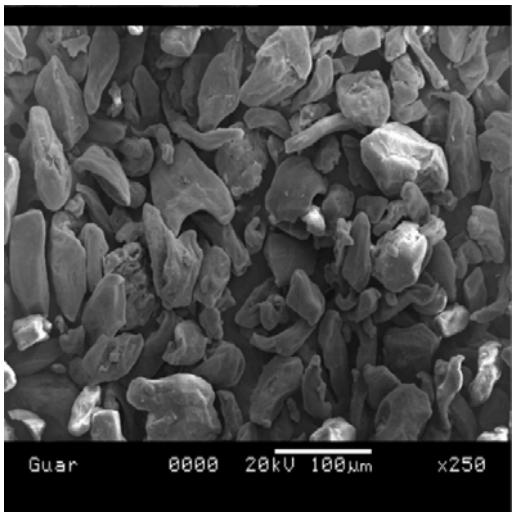

Fig 2: SEM image for guar sample

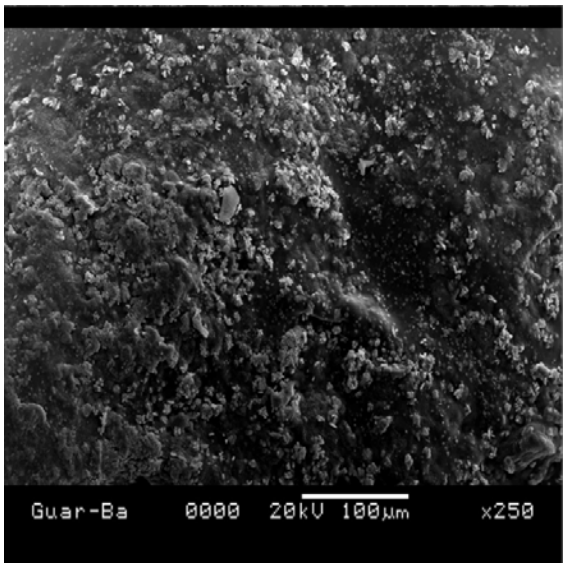

Fig 3: SEM image for guar – Ba^{2+} sample.

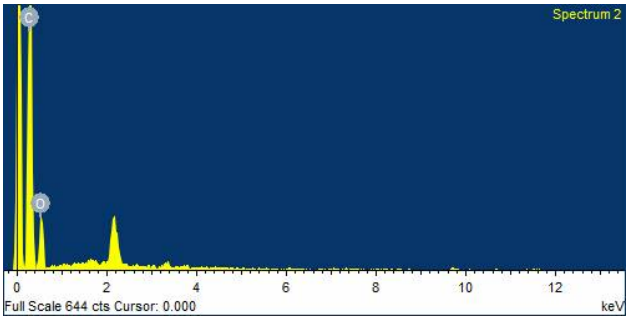

Fig 4: EDS study for guar sample.

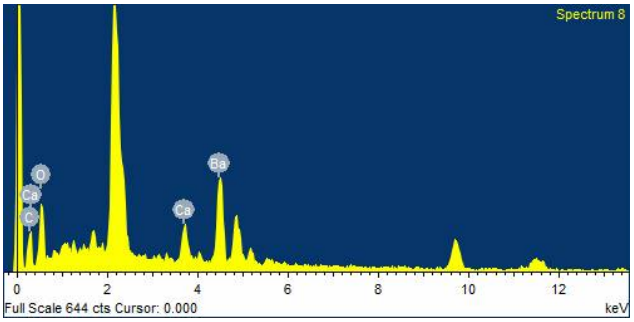

Fig 5: EDS study for guar – Ba^{2+} sample.

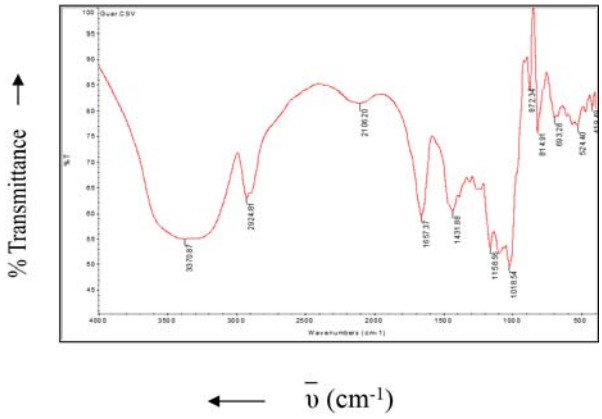

Fig 6: IR spectra for industrial guar sample.

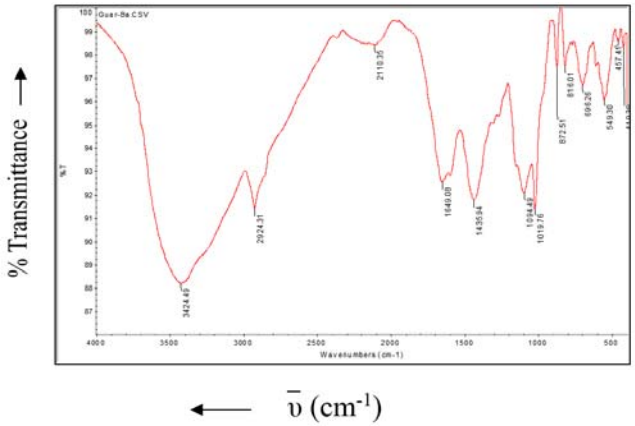

Fig 7: IR spectra for guar- Ba^{2+} sample.

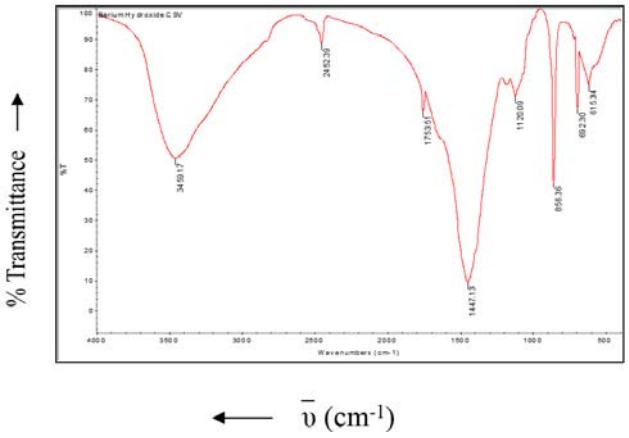

Fig 8: IR spectra for barium hydroxide.

4. Conclusion

Guargum and its derivatives are among the most important water-soluble polymers of industrial use. Recently $Y_1 Ba_2 Cu_3 O_{7-x}$ has been prepared by polymeric precursor technique. Binding of Y^{3+} and Cu^{2+} by guargum has been already studied. Present work provides valuable information regarding binding of Ba^{2+} by industrial guar sample. EDS study supports presence of Ba^{2+} in guar-Ba^{2+} sample SEM study supports welding of polymeric chains by Ba^{2+} in guar –Ba^{2+} sample. IR spectra supports binding of Ba^{2+} by –C-OH groups of guar sample at high pH.

5. Acknowledgement

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